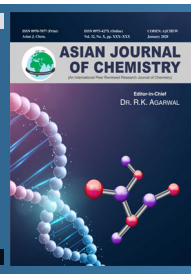
Asian Journal of Chemistry; Vol. 32, No. 12 (2020), 3043-3046

ASIAN JOURNAL OF CHEMISTRY

https://doi.org/10.14233/ajchem.2020.22884



Synthesis and Characterization of Nanoparticles of Iron(II) Gluconate Complex

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Received: 4 July 2020; *Accepted*: 9 September 2020; *Published online*: 7 December 2020; AJC-20137

This work describes the green synthesis and the characterization of nanoparticles of iron(II) gluconate complex, which is known to have antianemic properties. Iron(II) gluconate was synthesized by using ferrous chloride and sodium gluconate. The aqueous extract of leaves of *Cataranthus roseus* was used as a capping as well as reducing agent for the fabrication of nanoparticles of iron(II) gluconate complex. The synthesized Fe(II) complex and its nanoparticles were characterized by UV-visible, FTIR, SEM and FESEM techniques. Mechanism of plant leaf mediated synthesis has also been discussed.

Keywords: Nanoparticles, Fe(II) gluconate, *Cataranthus roseus*.

INTRODUCTION

The concept of nanotechnology is becoming innovative and revolutionary wherein already existing compounds are being modified as well as altered to nanoscale dimensions. As the size of materials proceed towards the direction of nanoscale, the properties of the materials get altered [1]. Nanoparticles of drug have been considered more advantageous in comparison with the conventional form of drugs [2]. For example, the surface area of a nanoparticle is very large, which provides special properties to the drug molecules such as high reactivity, high mechanical strength with enhanced electrical, optical and catalytic properties [3-5]. Moreover, nanoparticles of drug have extended half life time and takes longer time to circulate [5]. The organs and tissues are able to absorb the nanoparticle bound drugs more effectively and deeply through the skin.

The methods of synthesizing and characterizing the nanoparticles and its application in medical science and therapy have been reported in literature [6]. The synthesis of nano based particles can be done either by "bottom up" approach or "top down" approach [7]. In case of top down approach, there is a starting material and by reduction of its size, nanostructures can be created. The size reduction can be done by various physical and chemical processes [8]. In bottom up approach nanostructures can be produced from smaller entities such as particles, atoms or molecules. In this approach, firstly nanoparticles are created and then it aggregate to generate a final resulting particle [9]. Bottom up approach is considered to be more desirable in comparison with the top down approach because it produces the nanostructures with less imperfections or defects. There is also a better chance of production of short as well long nanostructures in bottom up approach [10]. Nanoparticles of any compound or drug molecule can be synthesized by plethora of chemical procedures, which include the usage of very reactive as well as toxic reducing agent which may have detrimental effects on the environment [11]. This has led to the need of alternate approaches for the synthesis of nanoparticles which are more reliable, eco-friendly and sustainable for the environment.

The plant extracts of eucalyptus leaves have been used to prepare Fe(II) complex nanoparticles in literature which did not involve the use of any harsh or toxic chemicals more stable as well as well functionalized [12]. In the overall synthesis, the construction of nanoparticles and usage of plant material in order to produce it generates a significant symbiosis between nanotechnology and plant based science. As a result, the integration of these two provides a greener approach in the field of nanotechnology termed as green nanotechnology. These processes are safe for environment and thus do not cause environmental pollution [13]. Biogenesis synthesis not only saves the environment but also produces nanoparticles in larger amount

with well-defined morphology and size [14]. Other environment friendly methods for the preparation of nanoparticles like use of microorganisms is also considered as green method but it takes longer incubation period whereas plant mediated synthesis is proven to be much better as it is very rapid, less expensive and scalable [15].

For the synthesis of nanoparticles by using leaf extract, the metal have to be mixed with the leaf extract [16]. There are numerous factors like the sort of phytochemicals, concentration of metals as well as phytochemicals, temperature and pH, which not only decide the rate of formation of nanoparticles but also inspect their stability and yield [17]. The extract of plant leaf have the tendency to reduce the metal ions as they behave as both a capping as well as a stabilizing agent [18]. Due to the presence of various phytochemicals *viz*. amides, flavones, sugars, carboxylic acids, ketones and terpenoids, which plays a major role for the bioreduction of nano based entities [19-21]. It is also noticed that protein biomolecules or carbohydrates present in plant extracts behave as a reducing agent and initiate the generation of metallic nano entities [22].

Iron(II) gluconate complex is of immense importance because of its anemic properties [23,24] and used as food additive [25,26]. In this work, an attempt is made to synthesize nanoparticles of iron(II) gluconate complex using aqueous extract of leaves of *Cataranthus roseus* as capping agent as well as reducing agent.

EXPERIMENTAL

Synthesis of Fe(II) gluconate complex: An aqueous solution of NaOH (30% w/v, 2 mL) was added to the solution of 5 g of $FeCl_3 \cdot 2H_2O$ in 10 mL of water solution at pH 5 to get the precipitates of ferrous hydroxide which was filtered, dried and added in a round bottom flask containing 100 g of sodium gluconate in 50 mL of distilled water and the reaction mass was heated to 75 ºC with constant stirring. The pH of the reaction mass was maintained at 11 by adding NaOH solution (30% w/v) .The mixture was then further heated at 105 ºC for 36 h in the oil bath after that the reaction mass was allowed to cool at room temperature. The desired Fe(II) gluconate complex was precipitated by adding 100 mL methanol under stirring. The complex was then filtered and washed twice with 10 mL of distilled water. Finally, the complex was dried in hot air drier at 50 ºC for 8 h. Yield: 25 g as brown coloured complex.

Preparation of aqueous extract of *Cataranthus roseus*: About 20 g of *Cataranthus roseus* leaves (sun dried for 4-5 hours) were taken in 150 mL of distilled water and the mixture was refluxed in a Soxhlet apparatus for 6 h. Finally, the extract was filtered and filtrate was stored in refrigerator at 15-17 ºC.

Preparation of nanoparticles of Fe(II) gluconate complex: Iron(II) gluconate (1.5 g) was added in 6 mL of water extract of *Cataranthus roseus* leaves and the reaction mass was centrifuged for 2 h at 5000 rpm. The supernatant was decanted and the resulting product was washed twice with 5 mL of ethanol. The nanoparticles of Fe(II) gluconate were obtained in the form of dark brown coloured precipitates.

RESULTS AND DISCUSSION

UV-visible studies: The samples of sodium gluconate, Fe(II) gluconate and nanoparticles of Fe(II) gloconate complex were subjected to UV-vis spectrophotometric studies by using Spectronic 200 spectrophotometer and the samples were scanned in the region of 200-800 nm. The UV-visible spectrum of sodium gluconate (Fig. 1a) shows an absorption band at around 200 nm which reflects a primary band arising from two types of transitions in the carboxylate anion of gluconate and that two transitions are $\pi \rightarrow \sigma^*$ and $\pi \rightarrow \pi^*$.

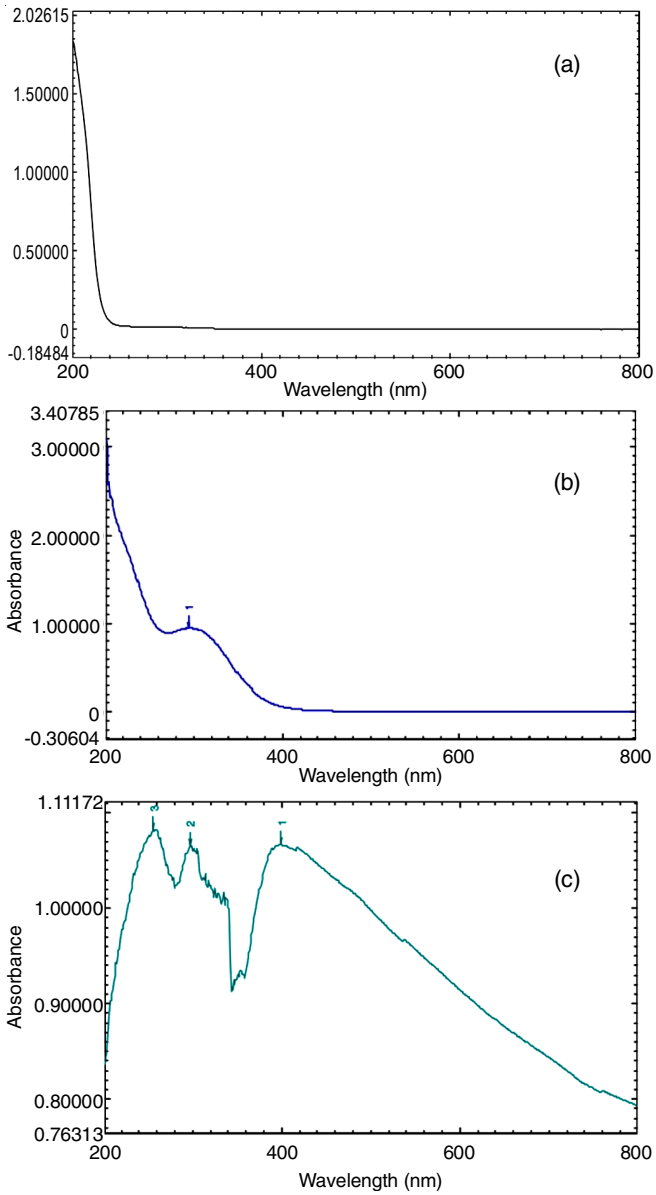

Fig. 1. UV-Vis spectra of sodium gluconate (a); Fe(II) gluconate (b) and nanoparticles Fe(II) gluconate (c)

The UV-Vis spectrum of Fe(II) gluconate (Fig. 1b) shows primary absorption 200 nm whereas secondary absorption took place at the range of 320-350 nm due to the resonance which occurs in carboxylate anions on reaction with ferrous ion. Moreover, this transition originated from $n \rightarrow \pi^*$ transitions of

the carbonyl functional group belonging to carboxylate anion due to the delocalization of the electrons in each carboxylate ion which are in the vicinity of ferric ion. The UV-visible spectrum of the nanoparticles of Fe(II) gluconate (Fig. 1c) shows the prominent band of iron nanoparticles (FeNPs) at around 250-260 nm.

FTIR studies: The FTIR (Nicolet iS5 spectrometer) spectrum of sodium gluconate is shown Fig. 2a. Band at 3537 cm^{-1} can be attributed to the primary hydroxyl group whereas band at 3427 cm^{-1} can be attributed to the secondary hydroxyl group. The peak of ν(OH) is visible at 1440 cm^{-1}, while the deformation vibration band of OH group at 646 cm^{-1} and the valence vibrations ν(C-O) of primary OH group appears at 1033 cm^{-1}. Moreover, the valence vibrations ν(C-O) of secondary OH group appears at 1093 cm^{-1}. Intense peaks at 1629 and 1041 cm^{-1} are also observed due to valence asymmetric along with symmetric vibrations of the carbonyl functional group of carboxylate anion whereas in the spectrum of Fe(II) gluconate (Fig. 2b) consists of one high intensity broad band peak at 3302 cm^{-1}, which is due to the valence vibrations of -OH originating

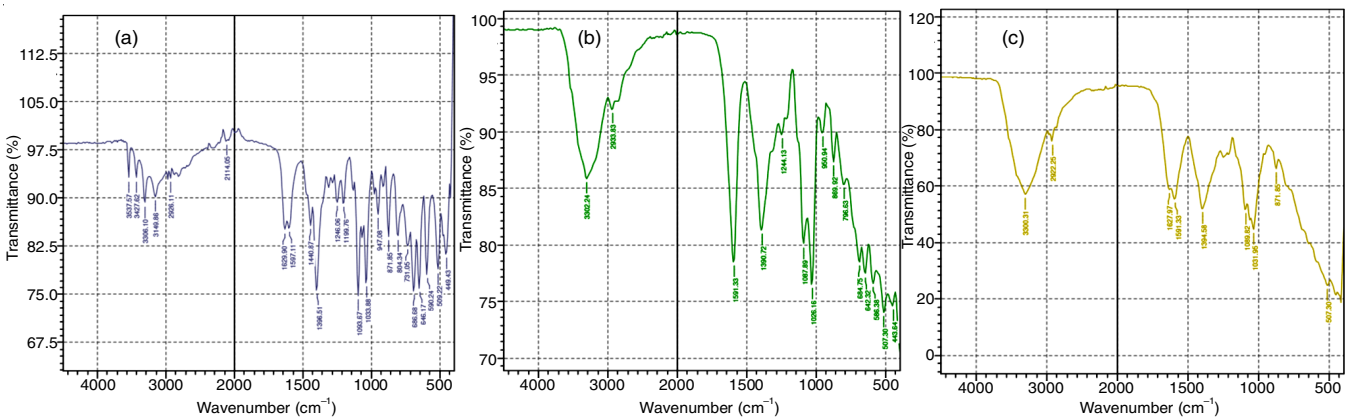

Fig. 2. IR spectra of sodium gluconate (a); Fe(II) gluconate (b) and nanoparticles Fe(II) gluconate (c)

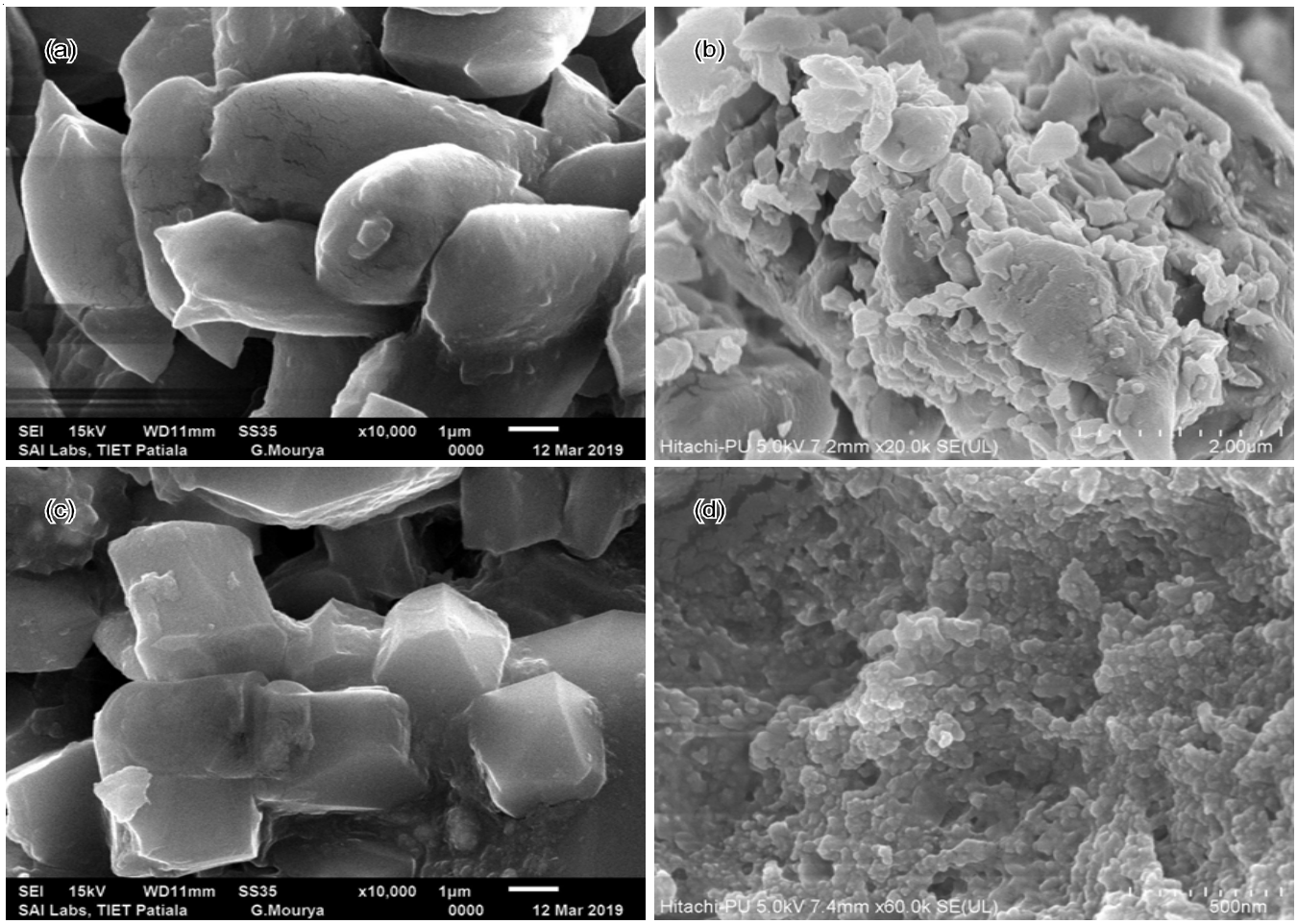

Fig. 3. Morphology of iron(II) gluconate complex [SEM (a) and (b) FESEM] and iron(II) gluconate nanoparticles [SEM (c) and (d) FESEM]

from H_2O present in the complex. In this area, the valence vibrations of primary and secondary -OH group of gluconate part can also be seen. Bands of deformation vibration of OH group in the plane as well as out of plane can be seen at 1390 and 642 cm^{-1}, respectively. Asymmetric and symmetric valence vibrations of carboxylate anion *i.e.* $\nu_{asym}(COO^-)$ and Fe-O vibrations appears at 1591 and 507 cm^{-1} respectively were also observed. Similarlly, the FTIR spectrum of nanoparticles of Fe(II) gluconate prepared from the leaves of *Cataranthus roseus* extract (Fig. 2c) shows the key adsorption bands at 3000 cm^{-1} ν(OH), 2922 cm^{-1} ν(C-H bond), 1628 cm^{-1} ν(C-C=C sym. *str.*), 1394 cm^{-1} ν(N=O bend), 1089 cm^{-1} ν(C-O), 1031 cm^{-1} ν(C-N *str.*), 871 cm^{-1} ν(C-Cl) and 507 cm^{-1} ν(Fe-O).

Morphology studies: The morphology studies of the prepared Fe(II) gluconate and its nanoparticles were performed using Hitachi, SUB010 instrument. The SEM micrograph demonstrates that the morphology of iron(II) gluconate complex were somewhat different and the surface was smoother as well as flatter, which reveals a uniform and tiny white flake embedded structure all over the surface of the synthesized complex (Fig. 3a-b).

The SEM micrograph signifies the cuboid pyramidal morphology of synthesized iron(II) gluconate nanoparticles at micro range (Fig. 3c). The FESEM analysis shows the aggregation of nanoparticles. In this micrograph, the observed spherical nanoparticles are in the approximate size range of 20-80 nm (Fig. 3d), which indicates the stabilization of the nanoparticles by a capping agent.

Conclusion

Iron(II) gluconate and its nanoparticles were successfully synthesized by using aqueous extract of *Cataranthus roseus* plant leaves. FTIR analysis indicates the possible functional groups which were involved in the reduction. Phenolic-OH group at 3000 cm^{-1} were noticed as a main bioactive phytochemical of *Cataranthus roseus* extract that acted as capping agent during the synthesis of iron nanoparticleswhich induces the stability of biogenically synthesized nanoparticles for longer time. The SEM analysis specifies the square pyramidal morphology of nanoparticles at micro scale. Only spherical nanoparticles having size in the range of 20-25 nm were observed at higher magnification, which was inferred from the FESEM analysis.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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